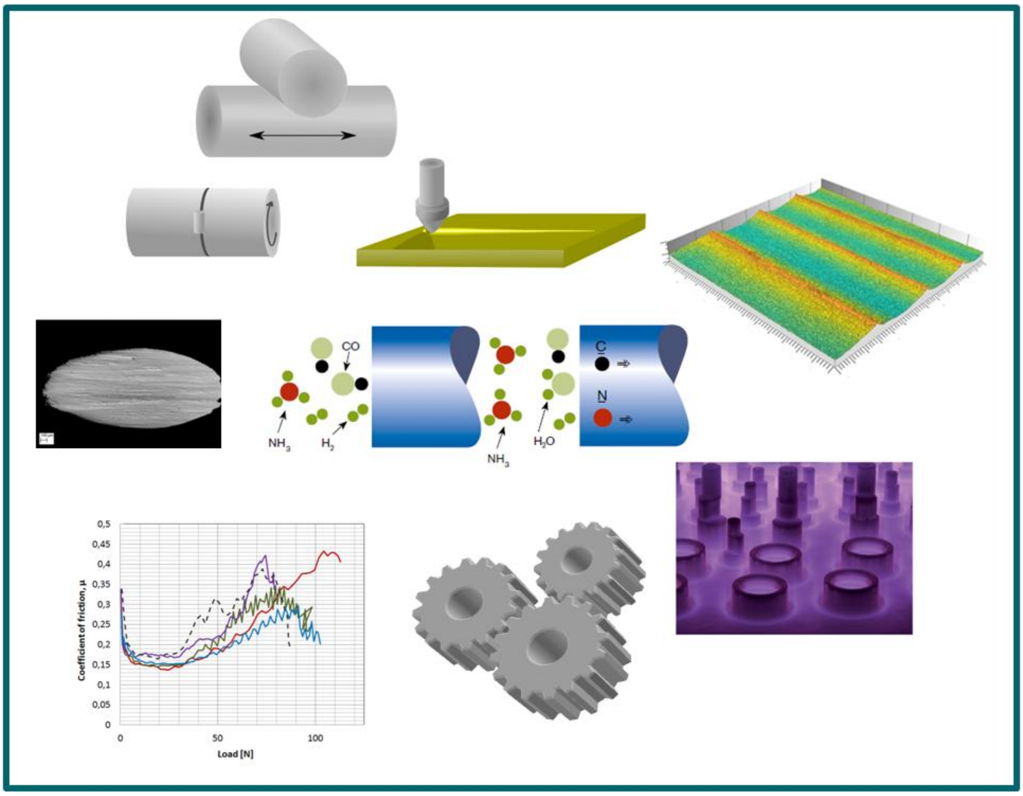
Surfaces with optimal friction and fatigue properties of nitrided/nitrocarburized components (SurfNit)

- Production, performance and sustainability aspects

Public report



Project within FFI - Sustainable Production Technology

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Content

1. Summary

Nitriding processes introduce a potential for reduced environmental impact both in production and during a product's life time. Surface properties, fatigue strength and corrosion resistance of a component can be improved significantly. Advantages in production are, compared to case hardening, less distortion and reduced energy consumption. During life-time of a component advantages are improved component performance regarding surface properties, improved fatigue strength and increased corrosion resistance. The properties can be further enhanced by post-treatments, eg post-oxidation and PVD coating.

In order to meet requirements for lower fuel consumption, low friction powertrain-components are needed. This involves components requiring high strength and excellent friction and wear properties. One purpose of the project has been to investigate the potential in developing and customizing the processes to create surfaces with high strength, low friction and excellent wear properties adapted for components such as gears and pistons. In the project the influence of compound layer properties e.g. hardness, porosity and phase composition on wear and friction has been studied. The tribological properties have been studied in a seizure test, a reciprocating sliding wear test, fretting test and a scratch test. The surface characteristic varies depending on steel grade combined with heat and post treatment resulting in different performance of the layer depending on test conditions.

To evaluate friction and wear performance properties selection of test method is highly dependent on the application. It was found that no single standard tribological test method can solely replicate practical knowledge of actual components, thus several tests in combination are necessary.
In the seizure tests and in the fretting tests all nitride/nitrocarburized behaved markedly better than the case hardened variant. The scratch tests showed that the ε-phase is much more brittle than the γ'-phase which could be undesirable in many applications. However the measured friction coefficient was identical in all cases ~0.1.
The variants with post processes (post oxidation and DLC Star coating) behaved outstanding in the seizure tests.
The surface topology is changed by nitriding/nitrocarburizing. Nitriding/nitrocarburizing adds a small isotropic structure on top the original topology so that a polished surface become rough while a rough surface does not change surface roughness values much. The surface roughness affects the wear and friction in sliding contact; smoother surfaces are typically associated in reduction of both wear and friction. In this study it has been shown that the surface roughness of the sample that wears least, whether it is because the material is more wear resistant or the total wear surface area is larger, is of higher importance than that of the counter surface. Therefore more focus should be on making the most wear resistance part of a material pair smooth, saving time and money on not preparing the counter surface as carefully. The influence of initial surface topography on the topography after heat treatment has been evaluated.

For a specific steel the ratio ε/γ´in the compound layer can be controlled by atmosphere (gas composition), time and temperature. Normally, carbon dioxide (and/or carbon monoxide) is used as a carbon source along with ammonia as a nitrogen source at nitrocarburization. However the nitrocarburizing process can be difficult to control and regulate due to high humidity in the atmosphere causing formation of e.g ammonium carbonate in the analyse equipment. The atmosphere is occasionally controlled by measurement of the hydrogen content to estimate the residual ammonia content. In the project a successful development have been made with other gas compositions in order to control the water content and thus facilitate the use of state-of-the-art IR-instrument for control and regulation of the process.

For steel Orvar Supreme, the fatigue strength increased by 10-15% for the tested nitrided processes, compared with through hardened test probes.

As a basis for the investigations, the requirements for selected demonstrator components were used. For demonstrator gear, an environmental evaluation was performed for nitrocarburizing and plasma nitriding with active screen compared to case hardening. The comparison was made with regard to energy consumption and does not take into account that another steel grade would be chosen for case hardening. The energy consumption for the two nitriding processes is approximately equal and about 33% lower compared to case hardening.

2. Sammanfattning på svenska

Idag är sätthärdning (uppkolning) den vanligaste värmebehandlingsmetoden för större volymer och högpåkända komponenter. Nitrering och nitrokarburering är ett av de mer intressanta alternativen. Nitrerprocesser möjliggör på flera sätt minskade miljöbelastningar, både i produktionsledet och under användningen samt ger ytterligare produktionsfördelar. Fördelar i produktion är betydligt mindre formförändringar och minskad energiförbrukning jämfört med sätthärdning. För användningssteget fås fördelar med förbättrad komponentprestanda med goda ytegenskaper (friktion och nötning), förbättrad utmattningshållfasthet och ökat korrosionsmotstånd. Efterbehandlingar som postoxidering och PVD-beläggning kan ytterligare förbättra egenskaperna.

För minskad bränsleförbrukning krävs drivlinor med låga friktioner. Detta kräver komponenter med hög hållfasthet och goda friktions och nötningsegenskaper. Ett syfte med projektet har varit att undersöka möjligheterna att utveckla och skräddarsy processerna (nitrering och nitrokarburering) för att skapa ytor med hög hållfasthet och friktions- och slitageegenskaper anpassade för komponenter som kugghjul och kolvar. Detta har gjorts och utvärderats i tribologiska tester där inverkan av stålsort, ingående yta och processparametrar vid värmebehandlingen har studerats. Inverkan av föreningszonens egenskaper som hårdhet, porositet och sammansättning (ε/γ′) har undersökts. Det saknas för närvarande en testmetod som i sig ger samma resultat som praktisk erfarenhet visar vid byte till nitrerprocesser. Det beror bl a på att friktion etc är väldigt beroende av applikation. Provningen har därför utförts i ett antal olika testmiljöer som bedömts vara de som är mest lika verkliga applikationer för de demonstratorer som används i projektet. Provningen har gjorts i ett skärningstest, fram-och återgående glidtest och ett reptest. Beroende på stål och värmebehandling varierar egenskaperna hos skiktet/ytan. Olika parametrars betydelse varierar beroende på provningsmetod.

Yttopografin har mycket stor inverkan när det gäller friktion och slitage. Inom projektet har studerats dels inverkan av olika ytfinhet på friktion och slitage, dels förändringen av yttopografin under nitrering/nitrokarburering.

Följande forskningsfrågor har studerats in om projektet:

- Vilkta standardtestmetoder kan användas för at studera skillnaderna i egenskaper efter sätthärdning och nitering/nitrokarburering som rangordnar på samma sätt som resultatet hos verkliga komponenter.
- Vilken sänkning av friktionskoefficienten kan förväntas vid ett byte från sätthärdning till nitrering/nitrokarburering?
- Hur förändras yttopografin vid nitrering/nitrokarburering beroende at utgångsytan?
- Kan föreningszonen skräddarsys till önskad sammansättning och tjocklek om gassammansättningen i ugnen mäts och sedan styrs till önskad sammansättning?
- Vilken sammansättning och mikrostruktur på föreningszonen ger optimala tribologiska egenskaper beroende på applikation

Studerade stål har varit 42CrMo4, Orvar supreme, Ovako277 (16CrMnNiMo9-5-2F), 34CrNiMo6, Ovako 225A (18CrMnMo8-4F), Ovako 495B (48CrMoNi4-10F) och 20NiCrMo2-2.

Ingen enskild testmetod kunde duplicera den praktiska erfarenhet som finns vad gäller byte från sätthärdning till nitrering/nitrokarburering utan resultaten i projektet visat att i de flesta fall behövs resultaten från flera olika testmetoder. Resultaten indikerar att egenskaper vid skärning kan vara en stor bidragande orsak till att nitrering/nitrokarburering ger så goda resultat hos verkliga komponenter. Skärning bör därför studeras vidare. Uppmätta friktionskoefficienter i samtliga tester och för samtliga prover, både sätthärdat och nitrerat/nitrokarburerat låg på 0,1. Dessa resultat indikerar alltså att det inte skulle gå att minska friktionen nämnvärt.

Praktisk erfarenhet säger att hög andel ε-karbid i föreningszonen ger bättre slitageegenskaper men reptestet av nitrokarburerat respektive plasmanitrerat Ovako 277 visade på ett mycket sprödare beteende för det nitrokarburerade provet som hade en hög andel ε-fas jämfört med det plasmanitrerade provet som hade en hög andel γ´-fas.

Yttopografin har en stor inverkan på friktions- och nötningsegenskaper vid glidande kontakt; finare ytor ger minskad friktion och nötning. I testerna framgick det att yttopografin hos den yta som nöts minst, antingen om det beror på att materialet är mer slitagetåligt eller att den totala ytan är större, har större betydelse än motgående yta. Det är därför viktigare att lägga större fokus på att göra den mest motståndskraftiga ytan jämn, för att kunna spara tid och resurser på den andra ytan. Inverkan av ingående ytas topografi på yttopografin efter nitrerprocesser har undersökts för flertalet stål och värmebehandlingar. Nitrerprocesser skapar en kortvågig isotrop struktur på ursprungsytan. Detta är speciellt tydligt för slätare ursprungsytor. Om ursprungsytan är väldigt slät blir ytan grövre när att den kortvågiga strukturen skapas. Om ursprungsytan redan har en någorlunda grov struktur innebär inte den nya kortvågiga strukturen att ytan totalt sett blir grövre

För ett visst stål kan förhållandet ε/γ´i föreningszonen styras (inom gränser för vad som är termodynamiskt/kinetiskt möjligt) genom atmosfärssammansättning, tid och temperatur. I normalfallet används koldioxid (ibland även kolmonoxid) som kolkälla tillsammans med ammoniak som kvävekälla vid nitrokarburering. Ytterligare en fördel med att använda andra gaser än koldioxid är ökade möjligheter att styra och reglera nitrokarbureringsprocessen med förbättrad processkvalitet som följd. Nitrokarbureringsprocessen är pga av den resulterande atmosfärens höga fukthalt samt bildningen av ammoniumkarbonat (bildas från restammoniak, vatten och CO_2 vid avkylning) svår att gasanalysera. Hittills har man nöjt sig med att hålla gasflöden/-mixen in i ugn konstant och ibland för ökad kontroll även mäta vätehalten och därigenom uppskatta restammoniakhalten. I projektet har lyckade försök gjorts med reglerade gassammansättningar för kontroll av vattenhalten som möjliggör mätning av t ex ammoniak, CO och CO_2 med ett standard IR-instrument och troligtvis är bildningen av

ammoniumkarbonat i analysutrustningen inte längre ett problem. Detta medför dels större möjligheter att skapa önskade skikt dels ökad processtabilitet och effektiv felsökning samt att faktiska processförhållanden kan dokumenteras.
Riktlinjer för atmosfärskontroll:

- Tillsätt CO till atmosfären för att förhindra bildande av ammoniumkarbamat i analysslangar.
- Använd gasanalysutrustning och mät halterna av CO, CO_2, H_2, N_2 HCN och NH_3.
- Beräkna nitrerpotential KN och kolpotential KC
- Styr ingående gassammansättning så att KN och KC hålls konstanta vid önskade värden.
    - Högt KN ger tjockare och mer porös föreningszon
    - Högt KC ökar anldel ε-fas
    - Lågt KC ökar andel γ'-fas och minskar risken för cementit-bildning i föreningszonen

För stål Orvar Supreme ökade utmattningshållfastheten, vid roterande böj-provning, med 10-15 % för de provade nitrerprocesserna jämfört med genomhärdade provstavar.

Som bas för undersökningarna har kraven för valda demonstratorkomponenter funnits. För demonstrator kugghjul gjordes en miljöutvärdering för nitrokarburering och plasmanitrering med active screen jämfört med sätthärdning. Jämförelsen gjordes med hänsyn till energiförbrukningen och tar inte hänsyn till att ett annat stål skulle väljas vid sätthärdning. Energiförbrukningen för de två nitrerprocesserna är i stort sett lika och ca 33% lägre jämfört med sätthärdning.

3. Background

Today case hardening is the most common heat treatment method for larger volumes of high strength components. Nitriding and nitrocarburizing are some of the more interesting alternatives. Nitriding processes allow in several ways reduced environmental loads, both at production and during the life time of the product in use. Besides, additional production benefits are provided; less distortion and reduced energy consumption compared to case hardening. During the service life of the product obtained benefits are improved component performance due to good surface properties (friction and wear), improved fatigue strength and increased corrosion resistance.

The performance of a nitrided component is influenced by base material strength (including residual stresses), diffusion zone (fatigue properties) and the compound layer (wear, tribological and corrosion properties). By a proper selection of nitriding process and steel grade the performance of the component could be further customized by achieving surface properties and strength profiles with optimal performance for a given application.

A major challenge for many components is how to obtain required strength properties, e.g as high fatigue strength as can be achieved by case carburizing. Fatigue tests performed on gear wheels have shown that the bending fatigue properties of nitrided wheels are highly dependent on the selection of steel grade. Compared to carburized steel fatigue strength equal to, or with a major increase, were obtained for plasma nitrided wheels in steel 42CrMo4, 18CrMnMo8-4F (Ovako 225A), Orvar Supreme (premium AISI H13) and Nimax compared to case hardened 16MnCr5 [1].

In many cases components with low friction and good wear resistance are desired, e.g hydraulic components, pistons, gear wheels and tools. Low friction powertrain-components are needed in order to meet requirements for lower fuel consumption. For these kinds of applications nitriding processes, combined with suitable steel, are of great interest.

Wear properties are affected by the hardness of the compound layer and diffusion zone, porosity and composition of the compound layer. A high ratio of ε/γ' in the compound layer is considered positive for improved resistance to wear under high loading [2].

To evaluate friction and wear performance the properties are very dependent on the application and thus the selected test method. However, a method to distinguish the performance of different layers is needed. In this project the tribological properties of nitrided steels have been studied in a seizure test, a reciprocating sliding wear test and a scratch test. The tests have been applied on different combinations of steel grades, nitriding processes and, in some cases, post treatments. Surface finishes, composition, thickness and porosity of the compound layer have been varied to be able to determine each parameter’s effect on the tribological properties.

The surface topography is essential when it comes to friction and wear. The surface topography will be influenced by the nitriding process and will depend on the topography of the original surface. Surface topography has been monitored throughout the project.

Nitrocarburizing exhibit quite a large scatter in the compound layer thickness and composition. One reason for this is that it is difficult to control and regulate due to the presence of CO_2 and high humidity in the atmosphere causing formation of solid ammonium-carbonate and carbamate in analysing lines and the equipment if attached. Therefore constant gas flows is the traditional way to secure a stable process. The atmosphere is sometimes controlled by measurement of the hydrogen content to estimate the ammonia content. A method not only measuring H_2 but also being able to measure the ammonia-, CO- and CO_2- content directly would facilitate increased possibilities to control and optimise the atmosphere depending on desired surface properties as well as improved process stability. Within this project a method to eliminate ammonium carbonate/carbamate formation is tested and experiments with carefully controlled atmospheres are performed.

4. Purpose, research questions and method

Purpose

The purpose of the project is to facilitate an increased use of nitriding and nitrocarburising for drivelines and to increase the performance of the heat treated components by developing and tailoring the processes to create surfaces with high strength, low friction and good wear properties for production of e.g drivelines with increased performance and minor losses. The aim is to achieve flexible production, reduced discards, lower fuel consumption and reduced environmental impact during manufacture and use of components.

To be able to compare and optimise the performance of different nitrided surfaces the friction and wear mechanisms needs to be better understood compared to e.g a case hardened surface.

For production and process engineering issues, the purpose is to develop and test guidelines for new atmospheres as well as better control and regulation of the process, which enables improved process control. Dimensional changes are considerably less after nitriding processes compared to case hardening, but they are not negligible. The size of these was to be determined.

The mechanisms of the nitrided surfaces during friction and wear needs to be better understood. In the case of wear and friction, compound layer properties e.g. hardness, porosity and phase composition (ε/γ'-ratio) are crucial. The performance of a nitrided component is influenced by base material strength (including residual stresses), diffusion zone (fatigue properties) and the compound layer (wear, tribological and corrosion properties). The mechanisms of the ceramic nitride surfaces compared to the metallic martenistic surface after case hardening is of interest since case hardening is the most common bulk heat treatment for high loaded components today.

Friction and wear performance properties are very dependent on the application. In this case the test matrix is extensive with many parameters and levels. For this reason a screening method, or several, to distinguish the performance of different layers is needed.

Depending on selection of steel grade, nitriding process and e.g post treatment the surface properties can be influenced to a great extent. Important parameters that are affected, and thus can be tailored, are:

- Composition of the compound layer; ε/γ'- ratio
- Influence of the porosity of the compound layer
- Thickness of the compound layer
- Hardness of the compound layer
- Topography of the surface – before and after the nitriding process
- Residual stresses

- Hardness of the substrate
- Influence of post treatments such as post oxidising and PVD-coating

Another important issue is the influence of lubrication. For minimized lubrication the ability of the surface to sustain lubricant loss is of importance.

In order to customize the surfaces during nitriding processes better regulation and control of the process is needed. The nitrocarburizing process can be difficult to control and regulate due to high humidity in the atmosphere causing formation of e.g ammonium carbonate in the analyse equipment. By developing the atmosphere composition and introducing better control of the water content standard IR-instrumens should be possible to use thus facilitating measurement of ammonia, carbon dioxide and carbon monoxide.

Research questions

The following research questions are to be answered within the project:

- What standard tribological test methods can be used to study the difference in performance of actual components heat treated with case hardening or nitriding/nitrocarburizing?
- What change in friction coefficient can be expected when changing from case hardening to nitriding/nitrocarburizing?
- How is the surface topology changed by nitriding/nitrocarburizing depending on the original surface?
- Can compound layer formation be tailored to desired composition and thickness if furnace atmospheres can be measured and the composition in the atmosphere is controlled?
- What composition and microstructure of the compound layer gives optimal tribological performance depending on application?

Methods

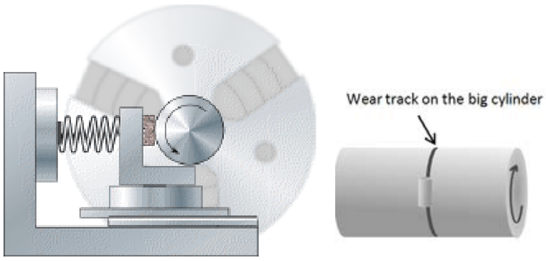
In this project a series of standard tribological test methods has been used. The choice of test methods was based on the applications of selected demonstrator components; a gear, a piston and a lance. None of the test methods replicates the actual component exactly but instead represents the closes possible alternative or best selection for evaluation of important properties.

Friction and wear testing

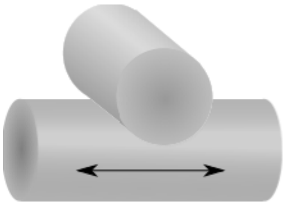
The *mechanisms* of the layer for friction and wear properties were studied using different test methods; a seizure test, a reciprocating sliding wear test, a fretting test and a scratch test, Figure 1. Test data were as follows:

- Seizure test
  - The seizure load was studied in a rig comprising a small stationary cylinder, 10 mm in diameter and with a length of 20 mm, pressed against a larger cylinder with a diameter of 40 mm, rotating with a speed of 900 rpm. The normal force was linearly increased, 7.5 N/s, using spring loading. The normal force was increased until the seizure threshold value was reached, here defined as a friction coefficient of ≥ 0.3, after which the turning machine was switched off. The rotating cylinder was pre-lubricated with a thin film of PAO oil. The film was applied by pouring a mix of 6 wt % PA08 in hexane and then letting the hexane evaporate. Before testing, all samples were cleaned in an ultrasonic bath, first in acetone then ethanol, 3 min respectively.
- Reciprocating sliding wear test
  - Reciprocal sliding between two crossed cylinders (10 mm Ø and 20 mm long), of the same steel was tested in a configuration where the upper cylinder is held stationary while the lower is mounted on a table, reciprocating with a stroke length of 4 mm. The applied load was 50 N, chosen to be high enough to cause noticeable wear scars after a reasonable number of cycles (100 000), while still low enough for the wear to stay within the compound layer. By doing this it is possible to study the effect of different microstructures in the compound layer. Before putting the cylinders in contact, a drop of base oil (PA08) was applied to the contact area, ensuring ample supply of lubricant during the whole test. The table was set to vibrate at a frequency of 30 Hz. The differences in surface roughness were reduced by a gentle grinding of the samples after the heat treatment.
- Fretting test
  - The fretting tests were performed in a similar equipment to the reciprocating sliding wear test and with identical test pieces. However, the stroke lengths were reduced to the order of microns instead of millimeters. Normal load was 9.81 and 19.62 N respectively. Lubricant was Shell Spirax S6 AXME 75W-90. Tests were performed with either a constant stroke length of 40 μm (peak to peak) or as interval with 500 cycles of 3 μm followed by 500 cycles of 40 μm. Total number of cycles 3 300 000 cycles per test.
- Scratch test
  - A Rockwell C diamond tip was used during the scratch test. The load was linearly increased from 0 N to 80 N, with a loading speed of 10 N/mm. No lubrication was used.

To be able to test many different material combinations in a reasonable amount of time accelerated testing was used. A fast screen test was first chosen to be able to get an overview of the different samples.



a)



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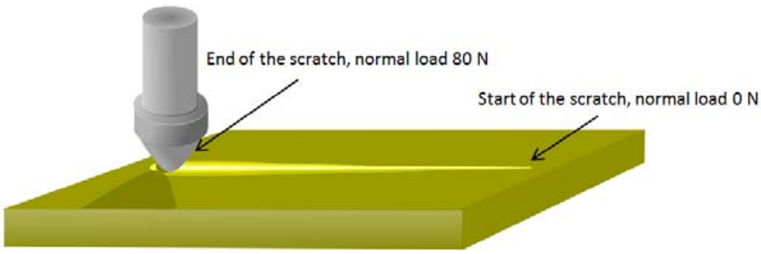


c)

Figure 1. Experimental set up for a) Seizure test, b) Reciprocal sliding test, in crossed cylinders configuration and c) Scratch test with a Rockwell C diamond tip (radius 200 µm) and a linearly increasing load.

The material resistance to brittle fracture was evaluated by using a scratch test. During this test the materials were subjected to plastic deformation and those materials that did not get cracks or delamination was thought as being tougher and less brittle.

Figure 2 shows the experimental set up for the fretting test.

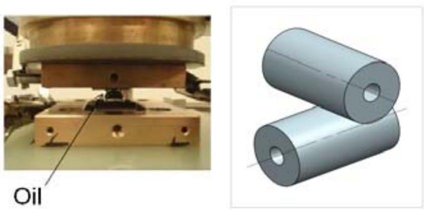


Figure 2. *Experimental set up for the Fretting test.*

The wear marks were studied in a high resolution FEG-SEM and chemically characterized using EDS mapping. The volume and depth of the wear scars was measured in an interference microscope as well as the original surface roughness. Interference microscopes uses the wave properties of light to measure height variations by comparing the path of two light beams, one reflected on a reference surface and one reflected on the surface of the sample. The surface hardness and the hardness of the substrate were measured with Vickers at a load of 1kg and 0.1kg as well as with nano indentation. Electron backscatter diffraction (EBSD) was used for microstructural characterization of the compound layer. EBSD is a microstructural crystallographic characterization technique for SEM using a phosphorous screen to detect backscatter electrons forming diffraction patterns. The symmetry and appearance of these patterns are related to the crystal structure of the sample.

Surface topography

The surface topography of the samples was measured before and after heat treatment. The influence of initial surface topography on the topography after heat treatment was evaluated. The aim was to study what influence the nitriding process had on the surface topography depending on its machined texture. An interference microscope (S neox from Sensofar) and software MountainsMap from Digital Suft was used to evaluate the surface topography of the steel samples before and after heat treatment. Measurement area was 880 x 660 µm with a lateral resolution of 0,26 µm. Form removal with 2'nd order polynomial and noise reduction was applied to the measured surfaces before analysis. Selected surface roufhness parameters were calculated according to ISO 25178-2 [3].

Studied steel grades

The chemical compositions of the steels studied are shown in *table 1*. The test probes had diameter 10 mm and were centerless grinded before heat treatment.

As a reference case hardened 20NiCrMo2-2 was included.

Table 1 Chemical composition, wt-%, of the steels.

Steel - Label	**C**	**Si**	**Mn**	**S**	**Cr**	**Mo**	**Ni**	**V**
42CrMo4 – A	0,38-0,45	0-0,40	0,60-0,90	0-0,035	0,90-1,20	0,15-0,30		
Orvar supreme – B	0,39	1,0	0,4		5,2	1,4		0,9
Ovako277 – C 16CrMnNiMo9-5-2F	0,14-0,17	0-0,030	1,20-1,40	0,023	2,10-2,30	0,45-0,55	0,45-0,55	
34CrNiMo6 – D	0,32-0,39	0,10-0,40	0,50-0,80	0-0,035	1,30-1,37	0,15-0,30	1,30-1,70	
Ovako 225A – E 18CrMnMo8-4F	0,16-0,19	0,20-0,40	0,75-1,00	0,005-0,030	1,75-2,00	0,50-0,60	0,30	
Ovako 495B – H 48CrMoNi4-10F	0,47-0,50	0,20-0,30	0,75-0,85	0,013-0,020	1,00-1,20	0,93-1,00	0,43-0,50	
20NiCrMo2-2 - G	0,18-0,23	0,20-0,35	0,65-0,90	0,008-0,040	0,40-0,70	0,15-0,25	0,40-0,70	

Heat treatments and post processes

All steel grades (except the case hardening20NiCrMo2-2) were preoxidized 1 h at 400°C and nitrocarburized in a standard nitrocarburizing of 580°C 150 minutes (Orvar Supreme 240 minutes).

In addition to this samples of Ovako 277 and Orvar Supreme were plasma nitrided at 540°C for 16 h, samples of Orvar Supreme and 42CrMo4 were coated with Balinit DLC Star after a short plasma nitriding (DLC Star Advanced).

Tests with post oxidised 42CrMo4 were also performed. Post oxidising means that the samples were oxidised by water vapour after nitrocarburising still in the furnace and at high temperature.

Residual stresses

The residual stress measurements were performed on a different set of samples than the rest of the project. Steels 46MnVS3, 42CrMo4 and Ovako 280 with three different compound layer thicknesses were studied.

Residual stresses were evaluated using XRD and a hole drilling method Prism (StressTech), which measures surface distortion using electronic speckle pattern interferometry (ESPI). In the XRD-analyse the peaks for ε and γ′ were used in order to analyse the residual stresses in the compound layer, Figure 3.

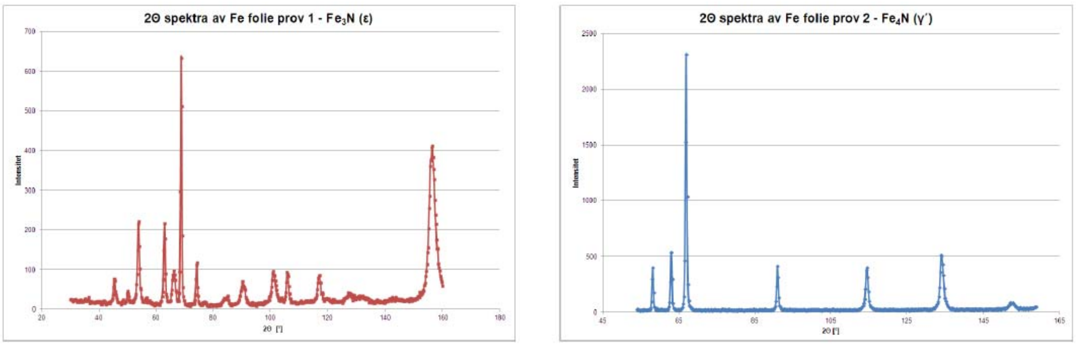


Figure 3. XRD-patterns for ε and γ′-phases

Fatigue strength

The fatigue strength was evaluated by rotating bending fatigue tests in a Roell Amsler UBM 200 machine. The test bar was clamped between two solid holders. The test bars were 120 mm long, 16 mm outer diameter and 10 mm diameter in the notch. The notch radius was 20 mm.

Process control

A new method to eliminate the formation of ammonium carbonate in analyse equipment has been developed in previous projects. When applied to production furnaces it showed that it was difficult to achieve the desired dryer atmospheres due to that post oxidation with water injection performed in production runs just before the tests. The tests were therefore performed in a laboratory furnace where the atmosphere could be closely monitored and controlled. Four different atmospheres with different nitriding and carburizing potentials were studied.

N_2, NH_3, CO, CO_2 and H_2 were fed into the furnace. The atmosphere in the furnace was measured by FTIR equipment and the flow of each gas into the furnace was adjusted to reach certain relations of partial pressures so that the desired nitriding and carburizing potentials were reached.

Influence on distortions and dimension

As a rule of thumb one third of the compound layer thickness leads to a dimensional increase. This was investigated for production components.

Influence on cost, environment and production aspects

A simplified LCA was performed to evaluate the environmental impact for the different processes used for the gear wheel. In this only the energy consumption for the heat treatment was included. All process steps before and after heat treatment were assumed to be equal.

5. Objective

The impact goal is to enhance an increased use of alternative environmentally friendly and cost-effective heat treatment processes for the production of drivelines with increased performance and minor losses. This to achieve flexible production, reduced discards, lower fuel consumption and reduced environmental impact during manufacture and use of components.

Project objectives:

- To introduce customized nitriding processes to achieve increased process/product quality, a more stable process, shorter process times as well as reduced distortions and scrap runs. Shape changes should be evaluated for some of the demonstrator components.
- For selected components show that it is possible to achieve a 25% reduction in friction or wear by 50% at boundary lubricated conditions, and also reduce friction by 50% in case of temporary lubrication losses, as compared to case hardened reference. This is done by choice of nitriding process, selection of steel, surface topography and e.g. post processes.
- Production aspects and environmental impact (LCA with focus energy consumption) are evaluated with a case hardened component as a reference.

Results from the project should be:

- Friction- and wear properties for tested steels, surfaces and treatments
- Guidelines for atmosphere and control/regulation of the nitrocarburizing process
- Guidelines for production regarding dimensional changes and how the surface topography before nitriding influences on the topography after nitriding.
- Recommendation of steel and heat treatment for the demonstrators.
- Friction and wear properties for post treated surfaces
- Published paper as part of doctoral thesis at the Ångström laboratory
- Cost, environmental, performance and production aspects for the demonstrators compared to case hardened component

6. Results and deliverables

How results and deliverables have contributed to the objectives of the FFI-program

Table 2 is a summary of the results from the project, deviations from results according to the application, and how they contribute to the objectives of the FFI-program.

Table 2 *Results to be delivered according to application, results from the project and contribution to the objectives of the FFI-program (according to the objectives from the application)*

Result to be delivered according to application	**Delivered**	**Contribution to objective of the FFI-program**
Friction- and wear properties for tested steels, surfaces and treatments	Results from seizure test, reciprocating sliding wear test, fretting test and scratch test has been presented. Steel grade and heat treatment influences on the result. The surface topography has a great impact on the result. However it is a big challenge to produce test samples with same topography since the nitriding process influences on the topography. The tested nitride surfaces had a better resistance to fretting compared to the case hardened steel. Further investigations are still needed to establish conditions suitable for minimized lubrication, tests with controlled topography......	The activity is aiming towards goal “30% mindre miljöpåverkan i tillverkningsprocesserna” and “Produktkraven m a p lägre vikt och ökad passiv säkerhet som i sin tur kräver nya eller förbättrade material och tillverkningsprocesser är uppfyllda”
Guidelines for atmosphere and control/regulation of the nitrocarburizing process	Tests have shown possibilities for monitoring and control of the furnace atmosphere using standard IR when the water content is reduced. By this tailored surfaces with controlled composition should be possible. Further research and testing is needed in order to implement this into industrial furnaces.	”Tillverkningsflexibilitet och framtagning av seriestorleksanpassade tillverkningslösningar i syfte att markant öka tillverkningsprocessernas och -systemens hållbarhet (ur ekologiskt och ekonomiskt perspektiv) har ökat” Nitriding processes are often used in batch furnaces with a higher flexibility compared to eg pusher furnaces

Result to be delivered according to application	Delivered	Contribution to objective of the FFI-program
Recommendation of steel and heat treatment for the demonstrators	Alternative nitriding processes, (one including a post treatment), compared to the treatments used in production today, were applied and evaluated for gear, a piston and a lance.	
Friction and wear properties for post treated surfaces	Post treated surfaces exhibited significantly higher seizure load and less wear compared to case hardened or nitrided surfaces	
Published paper as part of doctoral thesis at the Ångström laboratory	Paper to be submitted	
Cost, environmental, performance and production aspects for the demonstrators compared to case hardened component	LCA-energy → 33% less energy consumption for nitrocarburizing and plasmanitriding compared to case hardening. No other changes in the process flow was assumed. Influence of steel grade might need to be studied.	30% mindre miljöpåverkan i tillverkningsprocesserna

Surface topography, hardness and the microstructure of the compound layer

Table 3 shows compound layer thickness, porosity and surface hardness for the steels and heat treatments included in the study. When surface hardness is measured with HV1 an influence of the substrate will occur.

The nitrocarburized samples have a thicker compound layer with porosity compared to the plasma nitrided samples, Figure 4.

Figure 5 shows the EBSD images of steel 42CrMo4 nitrocarburized and steel Ovako 277 which has been nitrocarburized as well as plasmanitrided. Plasmanitriding of Ovako 277 results in a high amount of γ'. Nitrocarburizing of the steels results in a high amount of ε-phase.

Table 3 Microstructure and hardness of the samples included in the study.

Steel grade	Surface hardening	Post treatment	Compound layer thickness, µm	Surface Hardness HV1	Hardness HV0.1*	Porosity Compound layer %
42CrMo4	NC		13	744	618	9
42CrMo4	PN	DLC STAR	3.5	2288	636	-
42CrMo4	NC	Post oxidization	22	702	594	10
Orvar	NC		13	973	1059	8
Orvar	PN		4.5	1042	1019	0
Orvar	PN	DLC STAR	-	1645	315	-
Ovako277	NC		19	834	848	8
Ovako277	PN		7	-	809	0
34CrNiMo6	NC		12	819	601	6
Ovako225A	NC		15	829	716	8
Ovako495B	NC		19	740	599	3
Ovako152	C		-	857	729	-

*Measured 50 µm below the surface

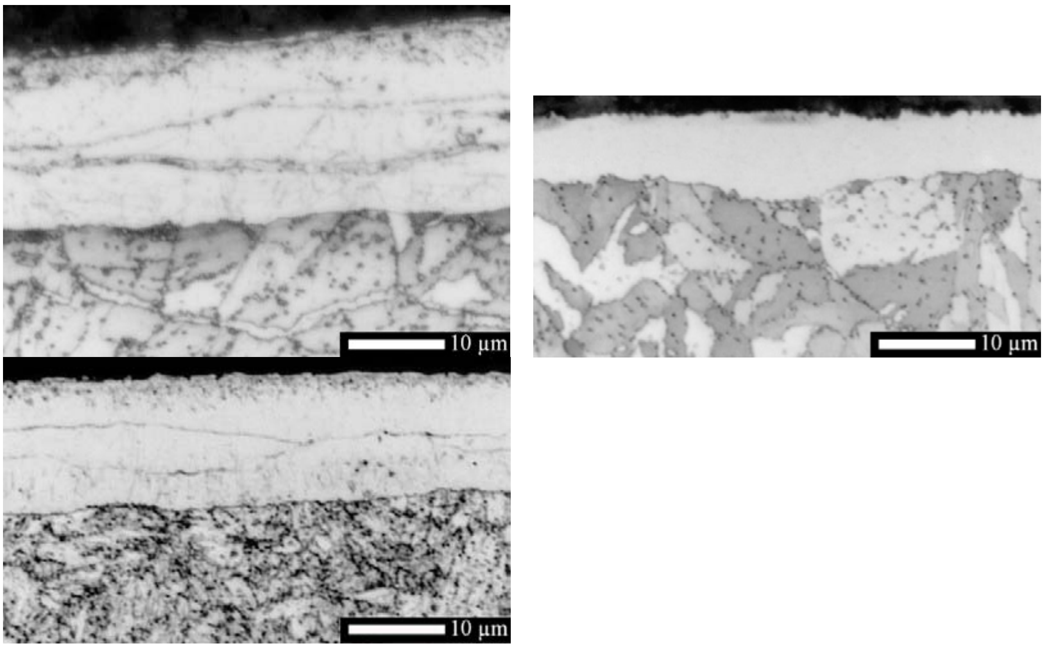
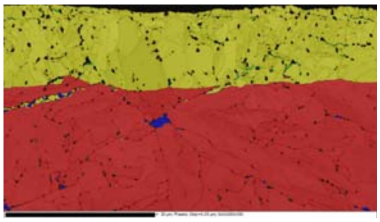
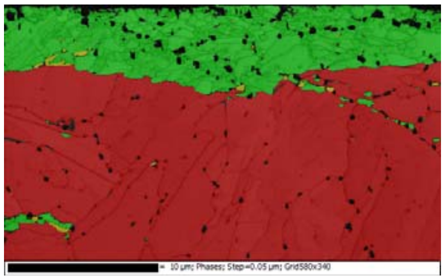


Figure 4. Cross section of the compound layer, LOM (light-optic microscope). To the upper left; Ovako277-NC, to the upper right; Ovako277-PN, to the lower left: 42CrMo4-NC.



a)



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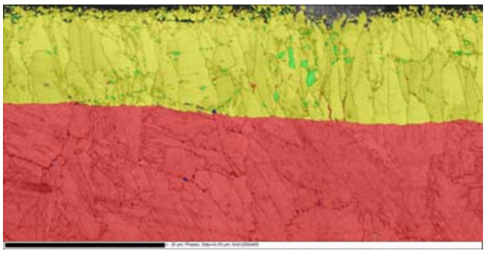


c)

Figure 5. EBSD images of steel a) Ovako 277 (16CrMnNiMo9-5-2F) nitrocarburized, b) Ovako 277 (16CrMnNiMo9-5-2F) plasmanitrided and c) 42CrMo4 nitrocarburized. (Note different scales)

For some samples the hardness was also measured with a nano indentor in the compound layer and diffusion zone. This was done for steel Ovako 277, nitrocarburized as well as plasmanitrided and nitrocarburized 42CrMo4. The hardness in the compound layer was at the same level for steel Ovako 277 nitrocarburized and steel 42CrMo4 nitrocarburized, both steels with a high amount of ε-phase. The lowest hardness in the compound layer was measured in steel Ovako 277 plasmanitrided, e.g. high γ´. Ovako277 plasmanitrided showed no hardness reduction between the compound layer and the diffusion zone in the first 100 µm from the surface, *Figure 6*. Ovako277 nitrocarburized has a higher hardness in the first 100 µm from the surface, including the compound layer.

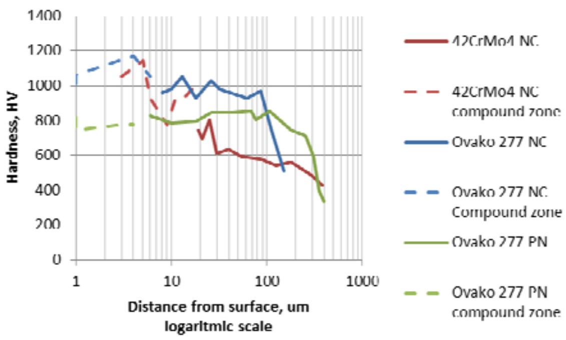


Figure 6. Nanohardness profile for steel Ovako 277 NC, Ovako 277 PN and 42CrMo4 NC.

Residual stresses

Figure 7 shows the residual stresses measured by XRD for a sample of 42CrMo4 with a compound layer of 21 µm. In this case the residual stresses were evaluated for the peak at 156° which is the peak for both ε-phase and martensite (it is not possible to separate the results for the between ε-phase and martensite). As can be seen the compound layer is subjected to tensile residual stresses and the diffusion zone is subjected to compressive residual stresses.

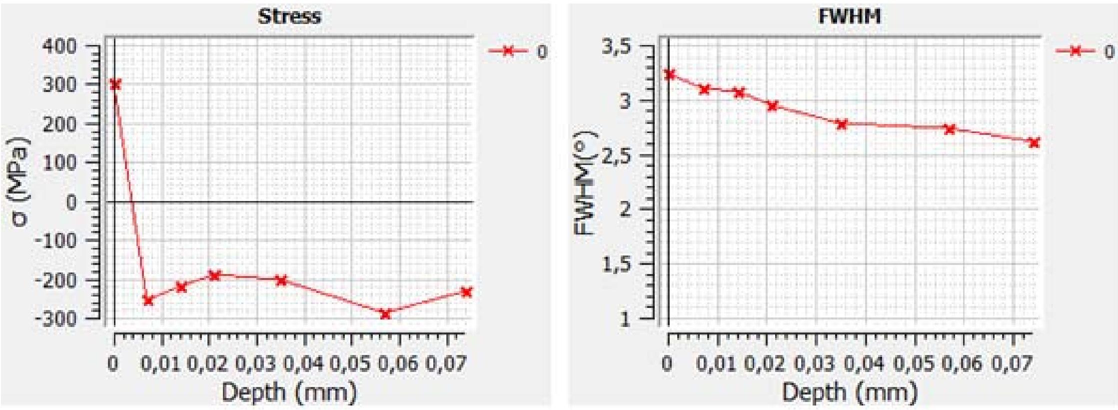


Figure 7. Residual stress and FWHM depth profile for sample A3. Peak fitting to ε-nitride and martensite peak at 156°.

Friction and wear testing

Seizure test

Figure 8 shows the result of the seizure test. The case hardened steel Ovako 253 exhibited the lowest seizure load while the post treated samples showed the highest. There is a small separation between the different steels. Most of the steels have a compound layer consisting of high amounts of ε and some γ', except Ovako 277 PN that has very high amount of γ′, and Orvar that has roughly 60% γ' and 40 % ε. Ovako 277 PN has a higher seizure load compared to the other. In the group of steels with high ε-content it´s hard to distinguish between the performance in the seizure test.

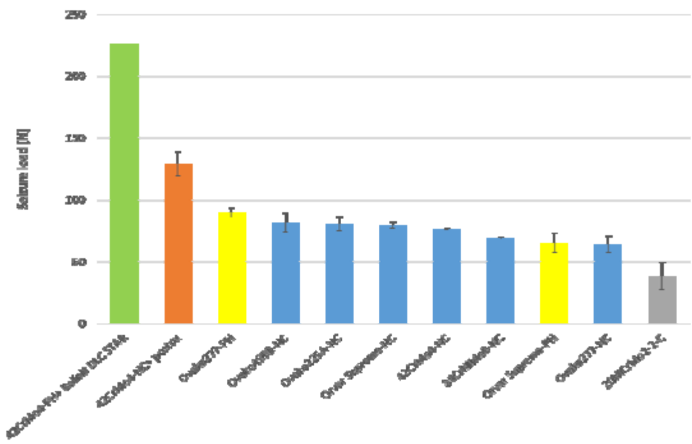


Figure 8. *Seizure loads of all tested materials. The bars represent the mean value and the error bars the standard deviation of the three parallel tests. Only one of three parallel tests for 42CrMo4-NC-DLC STAR Advanced reached the seizure threshold $\mu \geq 0.3$ and none of the tests of Orvar-PN-DLC STAR Advanced reached it.*

The influence of surface topography in the seizure test

When the counter surface is polished the friction and wear of the system is a lot lower than when the counter surface is grounded, see Figure 9. Since no seizure load was reached when the polished counter surface was used, all other experiments were done with the grounded cylinder.

There is no clear difference in the friction and seizure loads when the surface roughness of the small stationary cylinder is varied, Figure 10.

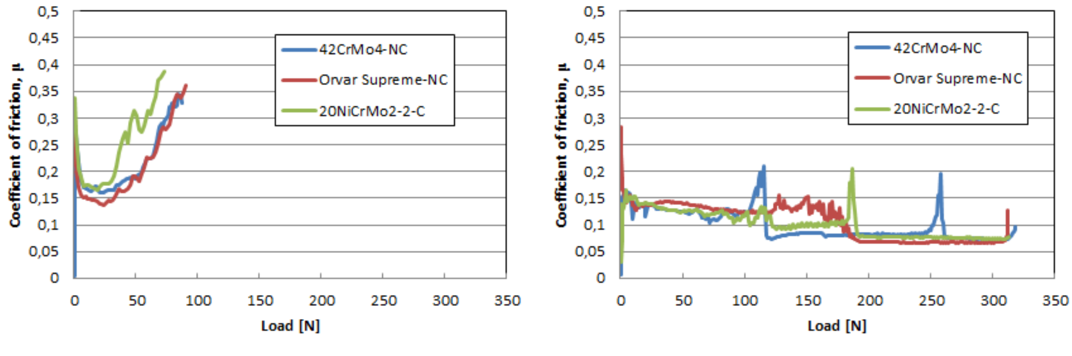


Figure 9. *Friction coefficient as function of load of three steels in the seizure test. Left: Ground counter surface. Right: Polished counter surface.*

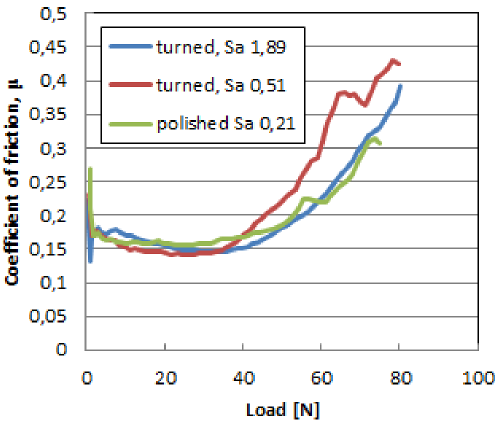


Figure 10. Friction coefficient as function of load in the seizure test. The same steel, 34CrNiMo6-NC, in the stationary cylinders, but prepared to different surface roughness.

Reciprocating sliding wear test

The result of the reciprocating sliding wear test is shown in Figure 11. Since the tested samples have different surface roughness and the amount of worn material is calculated from the measured volume of the wear scars, the values are estimations.

Although the standard deviation of the wear volume is relatively large for some of the samples within the same steel grade, there is a difference between the different steel grades. The depth of the wear scars on the stationary samples varied from 2 to 3 µm between the different steels. This means that the wear was kept within the compound layer for all the samples. The case hardened steel has the lowest average wear volume, but cannot be separated from the Ovako277 samples, both plasma nitrided and nitrocarburized. The two nitrocarburized steels show the largest standard deviation. One may note that of the three types on which hardness of the compound layer was measured with nanoindenter (Ovako 277 PN, Ovako 277 NC and 42CrMo4 NC) the wear volume does not correlate with compound layer hardness. The hardness of Ovako 277 NC and 42CrMo4 NC was similar and the hardness of Ovako 277 PN was lower. In the wear tests Ovako 277 PN and Ovako 277 NC were similar and the wear of 42CrMo4 was higher. Another thing one may note is that the standard deviation in wear volume of the nitrocarburized variants is much higher than that of the plasmanitrided variants. The standard deviation of the case hardened variant was even lower.

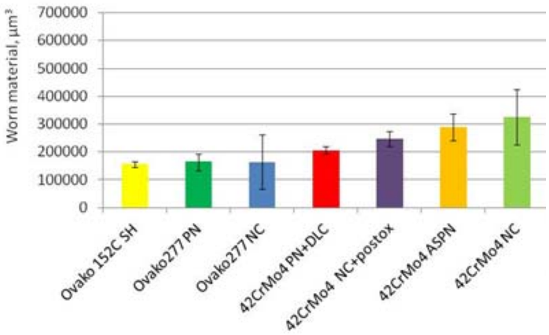


Figure 11. Wear of the stationary cylinders in the reciprocal sliding test. The bars represent the mean value of three parallel tests and the error bars show the standard deviation.

Fretting test

Figure 12 show the results of interval fretting tests with a load of 2 kg. All samples exhibited identical friction force for most part of the test. A nitrocarburized sample exhibited initial high friction for the first ~100 000 cycles which later dropped to the same values as the other samples. The plasma nitrided sample did not have this initial high friction, neither did the case hardened sample.

At higher loads the case hardened variant started running into problems after >2 million cycles when the friction force drastically increased to values same as a non-lubricated test. After another few hundered thousand cycles the friction force dropped again. This could be due to that wear of the surfaces produced perfect matching surfaces resulting in squeezing the lubrication out with a dry contact as a result. When the test continued the wear in the contact increased which later allowed new oil to enter the contact point, resulting in low friction.

At even higher loads test with case hardened samples were impossible to perform due to seizure whereas nitride/nitrocarburized test could be run with low friction. This could be another indicator that there is a difference in the seizure behavior between the two types of surface where a nitride/nitrocarburized surface performs much better than a carburized surface.

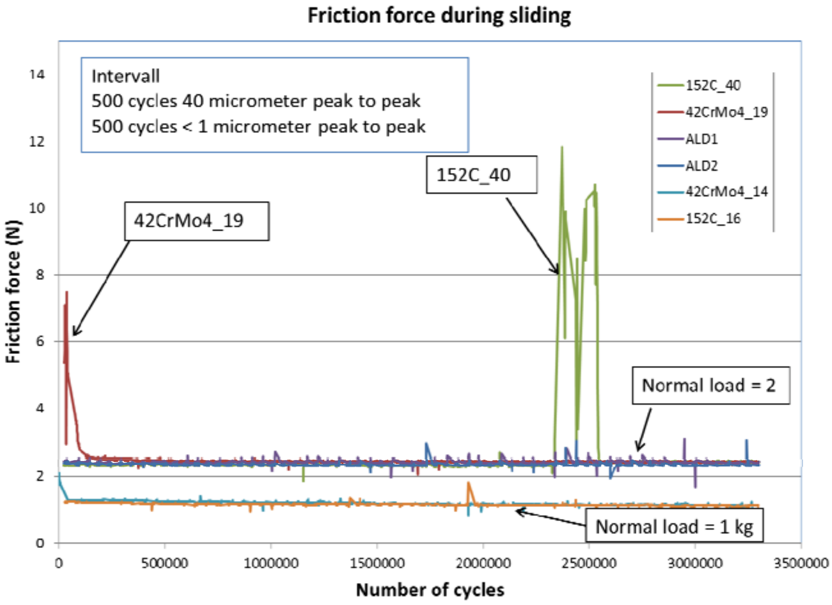


Figure 12. Results from the fretting tests. Interval tests,500 cycles 40 µm peak to peak followed by <1 µm peak to peak, load 2 kg. The figure shows the mean value of the friction force of every thousand 40 µm peak-to peak-cycle.

Scratch test

The scratches made in steel Ovako 277 and Orvar Supreme but with different heat treatments exhibit clear difference, Figure 13. The nitrocarburized samples, with a high ε-phase, have more brittle appearance, with the outmost layer flaking off outside the scratch. The plasma nitrided samples, with high γ′, have bigger ridges along the scratch and no cracks, indicating a more plastic behaviour. The case hardened sample has even higher ridges and is generally more deformed, Figure 14.

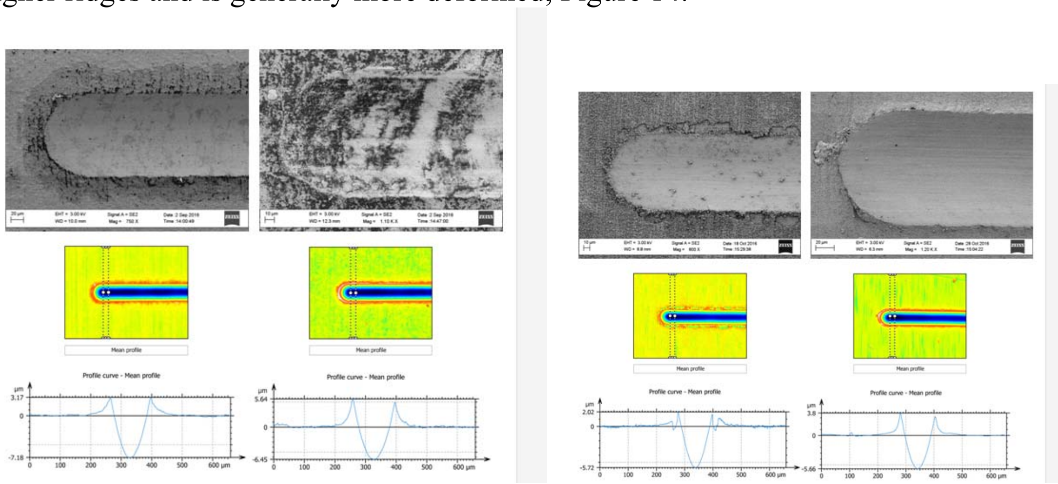


Figure 13. Appearance and depth profiles of scratch tested samples; one nitrocarburized and one plasma nitrided in left) steel Ovako 277 right) Orvar Supreme. The high load end of the scratch is shown. Left sample is nitrocarburised and right sample plasma nitrided.

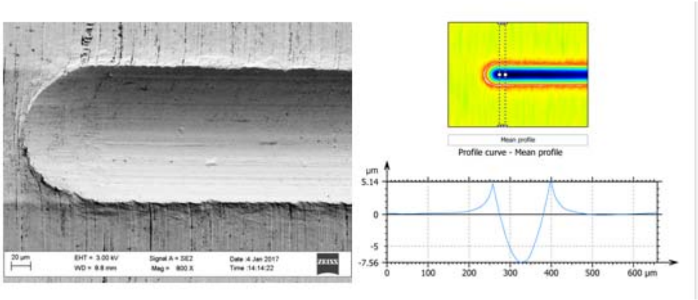


Figure 14. *The end of the scratch depicted and the depth of the scratch measured for Ovako152-C, the case hardened steel.*

Surface topography

For influence of surface topography two sets of surfaces were prepared:

- Flat samples, diameter 40 mm, in steel 42CrMo4, ground to different surfaces
- Cylindrical samples, diameter 10 mm, in steel 34CrNiMo6; turned to different Ra

Figure 15 shows surface topography for two differently ground areas on 42CrMo4 substrates. A and B is a rougher surface, with 80 grit final polishing step, before (A) and after (B) nitrocarburizing. C and D is a smoother surface, with 800 grit final polishing step, before (C) and after (D) nitrocarburizing.

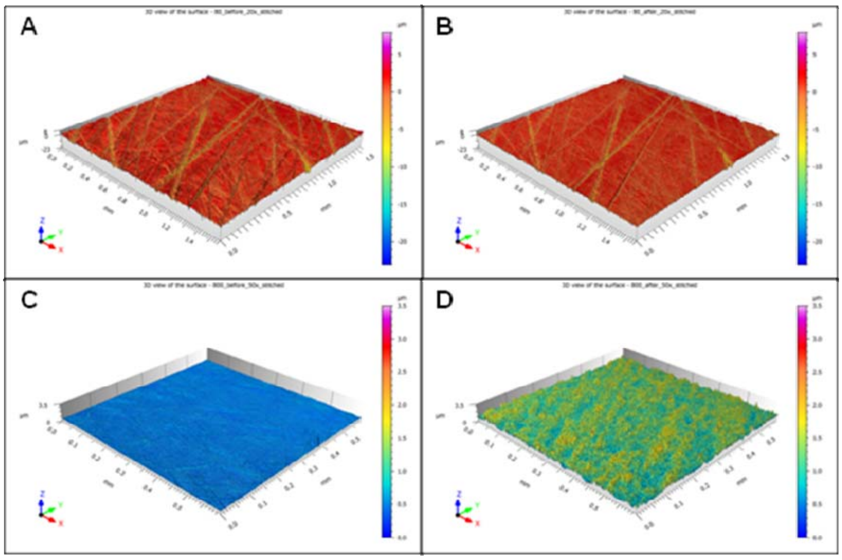


Figure 15. *Surface topography for two differently ground areas on 42CrMo4 substrates. A and B is a rougher surface before (A) and after (B) nitrocarburizing. C and D is a smoother surface before (C) and after (D) nitrocarburizing.*

Table 4 shows some surface characteristics for four different surfaces. Sa is arithmetic mean height of the roughness profile, Sdr is developed interfacial area ratio (surface area contributed by the texture compared to an ideal plane), Str is the texture-aspect ratio and is a measure of isotropic/anisotropic of the surface (1=completely isotropic and 0=completely anisotropic).

Table 4 Characteristics for prepared surfaces of steel 42CrMo4 before and after nitrocarburizing.

Surface	Sa, µm		Sdr, %		Str	
	Before	After	Before	After	Before	After
80	1,30	1,00	43,37	50,58	0,27	0,25
800	0,03	0,23	0,32	19,99	0,17	0,57
R0.4 Ra0.6	0,46	0,52	1,12	1,32	0,035	0,037
R0.8 Ra1.6	0,98	1,89	3,61	3,01	0,082	0,096

In general it can be stated that:

- The nitriding processes creates a short-wave isotropic structure on the original surface. This is particularly evident for smoother original surfaces.
- If the original surface is very smooth the surface will be rougher when the short-wave structure is created (Sa/Ra increases).
- If the original surface already has a fairly rough texture the new short-wave structure will not create a coarser surface (Sa/Ra is about the same).

Fatigue testing

The results of the rotating bending fatigue tests for different heat treatments of steel Orvar Supreme are summarised in *table 5*. Compared to through hardening the fatigue strength is improved 10-15% with the applied treatments. The difference between Balinit DLC and Balinit DLC Advanced is that the later one is plasma nitrided before the PVD treatment. This results in a slightly higher fatigue strength.

Table 5 Result of the rotating bending fatigue test of steel Orvar Supreme.

Heat treatment	Fatigue strength, MPa	Std. dev., MPa	Core hardness, HV1	Surf hardness @50 µm, HV0.1
Through hardenened, 49 HRC	842	31	540	562
Nitrocarb, 240 min	973	26		
Plasma nitrided	988	66	510	1230
Balinit DLC	938	19	543	539
Balinit DLC Advanced	955	26	550	598

Process control

The process control experiments were successful. It was shown that it was possible to measure the composition in the atmosphere without ammonium carbamate formation if the furnace atmosphere was kept dry enough. The experiments also showed that it was possible to control the atmosphere composition to reach desired gas partial pressure ratios and thus different compositions of the compound layer. If this technology would be transferred to industrial nitrocarburising furnaces the heat treatment results would be much more stable and predictable. The differences in the compound layers formed corresponded with the different atmospheres corresponded with what would be expected. Figure 16 shows compound layers formed on 42CrMo4 with atmospheres a)KN=4, KC=0.1 and b)KN=3, KC=0.05. Due to the lower nitriding potential in b) the compound layer is thinner and has lower amounts of γ' phase.

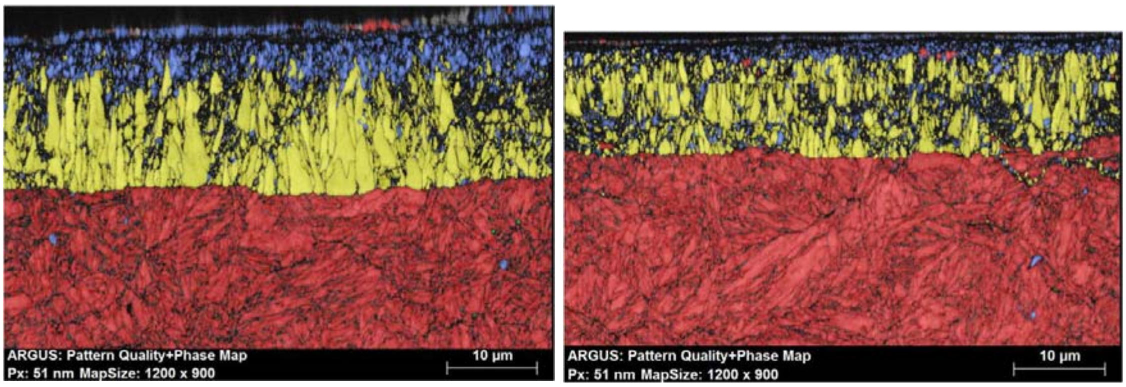


Figure 16. EBSD images of steel 42CrMo4 nitrocarburized with a) KN=4 and KC=0.1 b) KN=3, KC=0.05, yewllow ε-phase, blue γ'-phase, red ferrite

A literature survey was performed to find what in situ technologies to measure the furnace gas composition that are available on the market and could serve as cost efficient alternatives to the expensive FTIR instrument used for the experiments in this project.

Influence on distortions and dimension

Deviations from rule of thumb have been noted. Further research is needed in order to study the influence of heat treatment before the nitriding process. Recommendation is to do tempering at least 30 °C above nitriding temperature.

Demonstrators

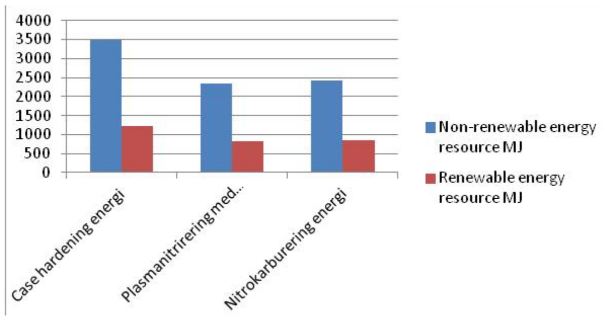
The demonstrators were a gear, a piston and a lance. These components are nitrocarburized today. The new alternative heat treatments that were selected for these components were:

- Gear. Plasma nitrided using active screen technique. Two different processes were used aiming for high and low amount of ε-phase. Dimension and surface characteristics were evaluated.
- Piston and lance. These were active screen plasma nitrided as well as coated with Balinit C Advanced. These components are planned to be field tested.

Influence on cost, environment and production aspects

For the cost comparison commercial heat treatment is considered. The cost for plasma nitriding is approx double as the cost for nitrocarburizing. This is probably also the case for in house heat treatment since the heat treating times will be longer for plasma nitriding.

Fel! Hittar inte referenskälla. a and b shows the result for demonstrator gear; comparing nitro carburizing and the new process plasmanitriding with active screen. As a reference also the result for case hardening is included. However in the last case another steel grade would be used, which is not considered in this study.



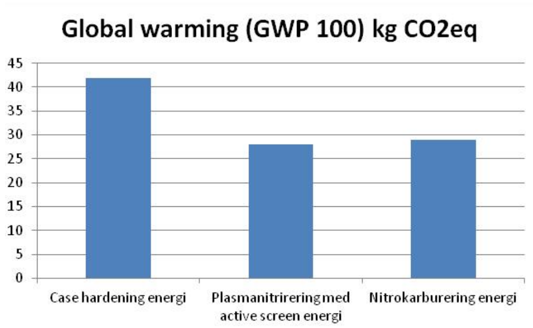


Figure 17. Comparison of energy consumption and CO2 equvivalents for case hardening, plasma nitriding and nitrocarburizing

The production flow for the gear would be the same for plasma nitriding and nitrocarburizing. The main difference would be longer heat treatment times.

7. Discussion

One focus of this work has been to facilitate an increased use of the beneficial properties achieved after nitriding and nitrocarburizing and to make the heat treatment process more stable and predictable so that they can be used to a greater extent. Also including areas where they are not so common used today, e.g. heavy-loaded vehicle drivelines.

It is known from earlier projects that highly stressed case hardened components can successfully be manufactured with a nitrided tool steel. It has been reported that the friction and wear properties is low for nitrided/nitrocarburzed components. It has also been reported that a higher ε/γ'-ratio is better than a low regarding wear properties. However, in several previous projects it has been difficult to replicate this, especially compared to a case hardened surface. In the previous project standard pin-on-disc tests have been performed, since it´s a convenient test method, even though it´s not directly related to any practical application. In the present project several different standard tribological test methods has been tested in order to determine a test procedure with good reliability and consistency with practical application depending on component. The methods were chosen for being the methods most relevant and closely resembling the demonstrators used in the project. It can be seen as a screening test to separate the properties of different combinations of steel and heat treatment. Also these methods did not fully replicate the practical knowledge that nitrided/nitrocarburized samples should behave better. The measured friction coefficient was identical in all cases ~0.1. It thus seems as it is not possible to lower the friction by changing to nitriding/nitrocarburizing. Also regarding wear standard case hardened samples behaved better than all nitrided versions. One reason for the poor results on wear could be that in all of the test methods wear particles from the brittle compound layer is still present in the contact area throughout the test for the nitride/nitrocarburized samples and may cause further wear, something that is not the case for a case hardened sample. However, in the seizure tests all nitride/nitrocarburized behaved markedly better than the case hardened variant. In the fretting tests the case hardened variant was not possible to test at high loads also indicating that seizure behaviour could be a major difference between the heat treatments. It could be so that the practical knowledge that nitriding/nitrocarburizing behaves better than case hardening is actually a result of the better seizure behaviour. In order to find a test method that fully captures the practical knowledge further studies of seizure behaviour is suggested.

The first research question was "What standard tribological test methods can be used to study the difference in performance of actual components heat treated with case hardening or nitriding/nitrocarburizing?" The project has shown that in most cases several tests are needed in order to compare the most important properties. No test method can be directly related to the environment for a real component. However, in order to minimize complicated and costly tests with real components screening tests are necessary. This project has brought much light on the differences between case hardening and nitriding/nitrocarburizing and on how to design a test that better replicates the practical knowledge.

It has been reported that the ε-phase is more desirable than the γ'-phase for wear properties. This could be due to the fact that the ε-phase is harder than the γ'-phase. However the scratch tests showed that the ε-phase is much more brittle which could be undesirable in many applications.

The variants with post processes (post oxidation and DLC Star coating) behaved outstanding in the seizure tests. Following the discussion above that seizure could be a significant parameter it is possible that both post processes would behave well in an actual application as well.

The second research question "What are realistic reductions in friction coefficients when changing from case hardening to nitriding/nitrocarburizing?" must, based on the results in this work, be answered with that at boundary lubricant conditions the friction coefficient seems to be the same for all tested systems. No large reduction is likely.

The third research question "How is the surface topology changed by nitriding/nitrocarburizing?" can be answered with that nitriding/nitrocarburizing adds a small isotropic structure on top the original topology so that a polished surface become rough. An already rough surface does not change surface roughness values much. The original surface has a great influence on the surface topography after nitriding processes.

The fourth research question was "What composition and microstructure of the compound layer gives optimal tribological performance depending on application?" This question is rather tricky to answer. The literature claims that a pore free ε-rich compound layer is better has not been duplicated in any of the tests in this work. The scratch tests showed better results for the softer and more ductile γ'-phase and the seizure tests showed both better properties for ε than γ' for one alloy and better properties for γ' than for ε for another alloy. The wear tests showed identical wear properties for ε and γ', for the single steel that was tested in both conditions. The answer to the fourth research question must thus be that more research is needed to determine what is an optimal compound layer.

The fifth research question "Can compound layer formation be tailored to desired composition and thickness if furnace atmospheres can be measured and the composition in the atmosphere is controlled?" can be answered with yes! The experiments showed that it is possible to control the atmosphere in the furnace carefully and that the compound layer formation then can be tailored to the extent that thermodynamics and kinetics allow for all specific steel grades. The method has yet to be implemented in an industrial furnace.

Guidelines for atmospheric control of a furnace are:

- Add CO to the furnace atmosphere to make it dry to avoid ammonium carbamate formation
- Add gas analysis equipment to the furnace so that CO, CO_2, H_2 and NH_3 can be measured in the furnace atmosphere
- Calculate ninriding potential KN and carburizing potential KC.
- Regulate incoming gas flows to keep KN and KC at desired values
    - High KN give thicker and more porous compound layer
    - High KC increases the amount of ε-phase
    - Low KC increases the amount of γ'-phase and reduce the risk of cementite in the compound layer.

8. Dissemination and publications

Dissemination

How are the project results planned to be used and disseminated?	Mark with X	Comment
Increase knowledge in the field	X	
Be passed on to other advanced technological development projects	X	
Be passed on to product development projects	X	
Introduced on the market	X	process control
Used in investigations / regulatory / licensing / political decisions		

Publications

Reports

Prel title: Nitrided and nitrocarburized steels for high stress tribological components; -influence on seizure limits from steel grade and surface treatment parameters. Viktoria Westlund, Ångström Laboratory. (To be completed)

Conferences and seminars

Properties of nitrided surfaces including post processes. Eva Troell, Johan Berglund, Swerea IVF; Sven Haglund, Swerea KIMAB; Viktoria Westlund, Ångström Tribomaterials group. European Conf on Heat Treatment 2016 And 3rd Int Conf on Heat Treatment and Surface Engineering in Automotive Applications. Prague, 11-13 May 2016.

Surface aspects of nitrided/nitrocarburized components – production and performance. Eva Troell[1], Johan Berglund[1], Sven Haglund[2], Jérôme Senaneuch[2], Viktoria Westlund[3]. [1]Swerea IVF, [2]Swerea KIMAB, [3]Ångström Laboratory, Tribomaterials Group. 5th Bodycote/AGA Heat Treatment Seminar. Material, Process and Innovation. May 20–21, 2014, Marina Tower, Stockholm.

Nitro carburized steels for high stress tribological components; -influence on seizure limits from steel grade and surface treatment parameters. V. Westlund; S. Jacobson. NordTrib 2016, 14-17 June 2016, Hämeenlinna, Finland

"Surface Topography of Nitrided Steel Surfaces".Johan Berglund, Swerea IVF.16'th Conference on METROLOGY and PROPERTIES of ENGINEERING SURFACES. 29 June 2017. http://www.metprops2017.se/

9. Conclusions and future research

In this work nitriding and nitrocarburizing has been studied with special focus on the properties of surfaces produced. The following can be concluded:

- To evaluate friction and wear performance properties selection of test method is highly dependent on the application. The best way is to do tests as close as possible to a field test. However, in this case a comparison of the surfaces for different steels and treatments was aimed for, thus demanding a screening test.
- No single standard tribological test method can solely replicate pratical knowledge of actual components, thus several tests in combination are necessary.
- The measured friction coefficient was identical in all cases ~0.1. It seems as it is not possible to lower the friction by changing to nitriding/nitrocarburizing.
- In the wear tests standard case hardened samples performed better than all nitrided versions. A likely reason for this is that particles from the brittle compound layer is constantly present in the contact area throughout the test for the nitride/nitrocarburized samples and may cause further wear.
- In the seizure tests and in the fretting tests all nitride/nitrocarburized behaved markedly better than the case hardened variant.
- The scratch tests showed that the ε-phase is much more brittle than the γ'-phase which could be undesirable in many applications.
- The variants with post processes (post oxidation and DLC Star coating) behaved outstanding in the seizure tests.
- The surface topology is changed by nitriding/nitrocarburizing. Nitriding/nitrocarburizing adds a small isotropic structure on top the original topology so that a polished surface become rough while a rough surface does not change surface roughness values much.
- Compound layer formation can be tailored to desired composition and thickness if furnace atmospheres are measured and the composition in the atmosphere is controlled.
- Nano-indenter is a useful instrument to measure actual compound layer hardness. No influence from the underlying substrate with this technique due to the small scale.
- The surface roughness of the sample that wears least, whether it is because the material is more wear resistant or the total wear surface area is larger, is of higher importance than that of the counter surface.
- Residual stresses were measured to be tensile in the compound layer and compressive in the diffusion zone.

Further research is needed in several areas to fully be able to use the full potential of the nitriding/nitrocarburizing heat treatments. Three main areas are identified.

- It was not possible to fully determine how an optimal compound layer should be constituted within this project. Further research is needed, possibly using more adequate test methods and strongly correlated to a certain application.

- Seizure properties seem to be one of the more significant and overlooked differences between a case hardened and a nitride/nitrocarburized surface. It is possibly this property that is the main reason of the outstanding performance of nitride/nitrocarburized surfaces. More research is needed in this field and alternative test methods are probably needed that more specifically addresses this property.
- The tests with atmosphere control showed that improved process control and tailored compound layers are possible. However this has yet to be implemented in an industrial furnace. In addition to this more research is needed in order to understand what compound layers are thermodynamically possible to tailor for a certain steel alloy. A PhD project is suggested for this.

10. Participating parties and contact persons

Project partner	**Contact person**
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Atlas Copco Secoroc	Richard Johanson
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Stresstech	Per Lundin
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Volvo CE	Joakim Johansson
Volvo Materialteknik	Ulla Boman
Volvo Powertrain	Linus Gustafsson
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